



OPEN Nano-Bio Soil Improvement (NBSI) with Micro-, Nano-, and Pico-*Typha latifolia*: a green material approach

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Soil stabilization is a method of improving weak soils by adding different additives. Nano additives and materials represent a new technology and a revolution in soil mechanics and geotechnical engineering, used for soil stabilization, a branch of soil improvement. This research evaluates the particles of Nano- and Pico-Typha solution as a new biomaterial for soil improvement using nano and pico scales, bio, and soil mechanics tests. The research investigates the changes in soil mechanical properties after the addition of Nano and Pico Typha solution as a Nano-Bio Geotechnics (NBG) technique, comparing the properties of the soil before and after stabilization. To control and characterize the size and nature of the particles studied in this research, SEM tests were performed after the nano production process. To check the particle dimensions and structure before and after the nano production process, XRF and XRD tests were performed. After converting particles from micro to nano scale, there are also smaller pico particles. The research studied the properties of Khavaran clay soil by adding Typha and Nano Typha as a Nano-Bio additive in different percentages of 3%, 5%, and 7%, and in curing times of 1, 7, and 28 days. The results showed that the uniaxial resistance of clay increased from 50 kPa to 12 times its initial value by adding 7% Nano-Typha after 28 days of curing. The maximum deviator stress of the soil increased by 10.1, 14.07, and 15.9 times its initial value in confining stresses of 100, 200, and 300 kPa, respectively, by adding 7% Nano-Typha after 1 day of curing. The cohesion and friction angle of the soil stabilized with 7% Nano-Typha solution increased by 2.22 and 6.3 times, respectively, compared to clay soil after 1 day of curing.

Keywords Nanotechnology, Clay, *Typha latifolia*, Nano *Typha latifolia*, Soil improvement, Nano-bio soil improvement (NBSI), Nano-bio geotechnics (NBG)

Soil stabilization plays a crucial role in geotechnical engineering and ground improvement. It involves altering the characteristics of soil at a site to make it more suitable for construction, addressing unsuitable and weak soils¹. Soil has been used in various industries and historical buildings across different countries, making the improvement of its engineering characteristics highly effective in soil mechanics². One method to enhance soil is by adding cement or chemical additives. These additives, ranging from macro to micro scales, include cement, bitumen, lime, and volcanic ash. They are primarily used to stabilize fine-grained clays and silts. Volcanic ash can reduce permeability and erosion while increasing the durability of granular soils. Additionally, it can reduce cement consumption and improve the soil's resistance properties^{3–5}. Nanomaterials possess unique characteristics that distinguish them from larger-scale materials, significantly influencing various engineering branches due to their distinct physical properties. In geotechnical engineering, nanomaterials are among the most advanced techniques for soil mechanics⁶. The substantial investment in nanotechnology research at a macro level underscores its growing importance in developed countries, with approximately 5.5 billion dollars invested annually in this field worldwide⁷. Geotechnical engineering stands to benefit greatly from integrating nanotechnology with soil mechanics, particularly in modifying the behavioral parameters of different soils⁸. Eyo et al. found that treating clay with RoadCem (RC) nanoadditives reduced porosity and improved properties⁹. Niu et al. reviewed cement-based materials with nanoclay, noting enhanced properties¹⁰. Garg et al. studied the

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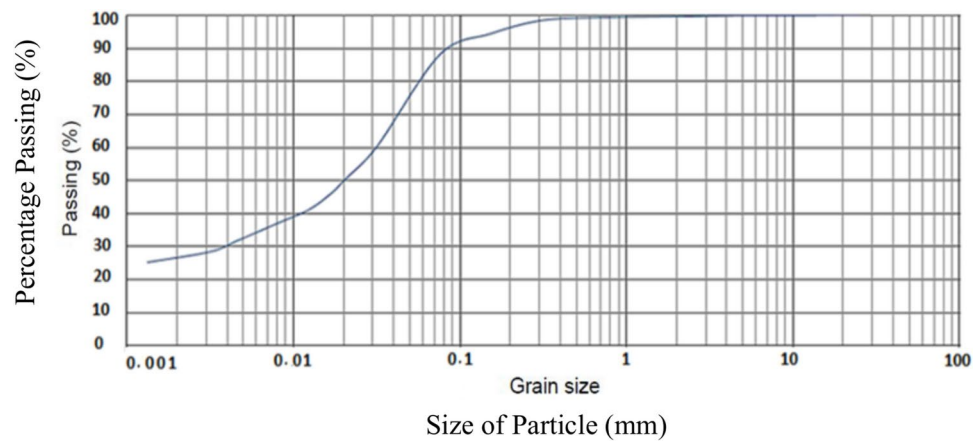


Fig. 1. The Grain size distribution curve of the studied soil.

LL	32
PL	15
PI	17
Compressive characteristics of Khavaran clay soil	
Maximum dry density	1.94 gr/cm ³
Optimal water content	11.8%

Table 1. Soil characteristics.

effects of nanoadditives on cement strength and structure¹¹. Selvakumar et al. and Kulanthaivel et al. examined the impact of various nanomaterials on clay and sandy soils, highlighting improvements in strength and properties^{12,13}. Sarooj, an ancient material used in Iran and some Persian Gulf countries, exemplifies traditional soil modification techniques². Although its exact date of use is difficult to determine, some examples in Iran are over 700 years old. Until the early 1990s, Sarooj was widely used in the Zagros region as the primary mortar for building stone walls and plastering interiors. Some Sarooji houses from this period still exist in Sar-Maleh village, Arghun Dashtestan, Bushehr province¹⁴. Sarooj production involved either a hot or cold method. The hot method included pounding clayey limestone clumps until soft, adding straw and water, spreading the mixture to dry, and then baking and grinding it into a light brown or lemon-colored product known as Hot Sarooj¹⁵. The cold method combined lime, ash, sand, clay, and *Typha latifolia*, an aquatic weed found in wetlands, swamps, and riverbanks^{16,17}. To enhance soil properties, adding specific additives is essential. However, current research literature lacks comprehensive studies on the behavior of *Typha* as a biomaterial and Nano-*Typha* particles as a Nano-Bio technique in soils. This research aims to address this gap and advance understanding in this area.

Materials and methods
Soil sample

Figure 1 shows the result of test (sieve-analysis test) and hydrometry for Khavaran clay located in Tehran according to ASTM D422¹⁸ standard.

ASTM D431 standard has been used to test the liquid limit and plastic limit on the studied soil. The Atterberg limits are essential for understanding the behavior of fine-grained soils. The soil sample has a Liquid Limit (LL) of 32%, indicating it transitions to a liquid state at this moisture content. The Plastic Limit (PL) is 15%, where the soil begins to exhibit plastic behavior. The Plasticity Index (PI), calculated as the difference between the LL and PL, is 17%, showing the range over which the soil remains plastic. These values suggest that the soil has moderate plasticity, which is crucial for soil classification and engineering applications. The results in Table 1 show that the studied soil is a clay soil with low plasticity (CL).

ASTM D854¹⁹ standard is used to determine the density of soil grains. According to this test, the specific gravity (GS) of soil equals to 2.65. ASTM D1557²⁰ standard has been used for modified compaction test. The output results for the studied soil can be seen in Fig. 2 and Table 1.

Specifications of the nanomaterials

Typha latifolia and Nano *Typha latifolia*

Nano-powder is the term for nano-materials in powder form. Their grains have one or three dimensions between 1 and 100 nm. Their chemical activity increases significantly as their particle size approaches 10^{-9} m. This makes the materials more plastic and gives them new and useful properties. Nano-materials can be used in soil stabilization in both dry and solution forms, but the solution form is more efficient than the powder form.

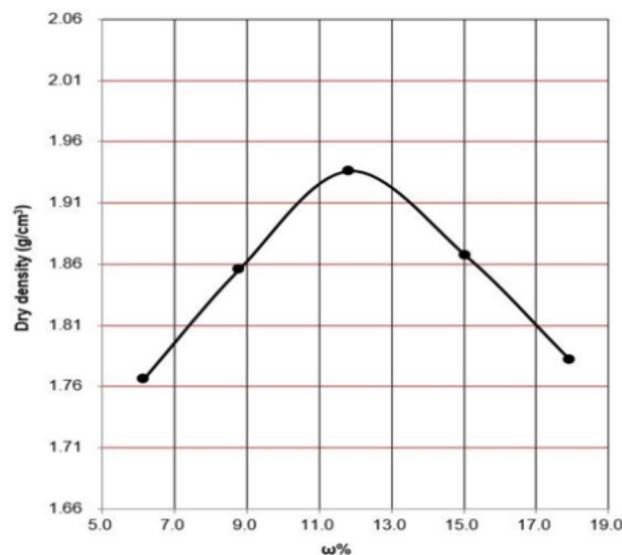


Fig. 2. Compaction curve of Khavaran clay soil.



Fig. 3. *Typha latifolia*.

Typha latifolia (Fig. 3) is a weed that grows in coastal and marshy areas. In Iran, there are about six species of this genus in fresh and shallow waters on the banks of rivers, wetlands, lakes and stagnant waters. The *Typha latifolia* used in this research was obtained from Mazandaran province in Iran. To produce the nano-particles of *Typha latifolia* (Nano-Typha), only *Typha latifolia* itself was used without any additives. This method of producing Nano-Typha is organic and efficient. The quality of the material depends on the size, purity and shape of the primary powder particles. The dimensions of Typha plant used in this study are about several tens of centimeters. The nano solution used in this research was prepared according to the invention of Nano-Typha solution²¹.

Preparation of the samples for experimental tests

To study the effects of stabilizing materials on the soil, two types of tests have been performed: soil mechanics tests and tests to determine soil composition and structure. The soil mechanics tests that can be done in unsaturated conditions are Unconfined Compressive Strength (UCS) tests (uniaxial) and unconsolidated undrained triaxial tests. These tests measure the structural resistance of the soil for geotechnical projects.

UCS test

The uniaxial test was performed according to the ASTM-D2166²² standard for cohesive soils. A two-piece sampler with a diameter of 38 mm and a height of 76 mm was used in this study. The samples were prepared with Nano-Typha solution at 3, 5 and 7% weights and with the optimal moisture content and the maximum dry weight obtained from the modified density curve. The samples were tested at different curing times of 1, 7 and 28 days. To maintain optimal moisture, the samples were stored in a plastic bag according to curing time.



Fig. 4. Sample tested in the triaxial (UU) device.

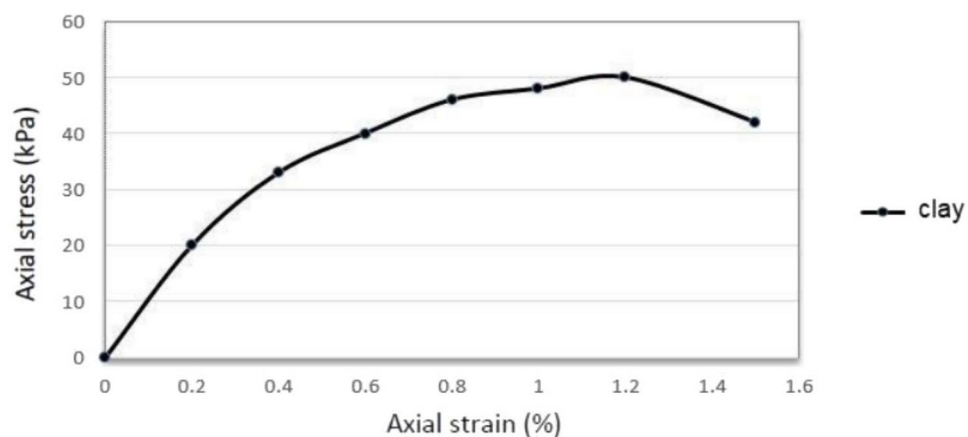


Fig. 5. Stress-strain curve of Khavaran clay soil.

Triaxial test

The triaxial test in undrained and unconsolidated conditions was performed according to the ASTM-D2850²³ standard. A two-piece metal mold with a height of 76 mm was used to prepare the samples. The clay soil was divided into 8 equal parts and pounded layer by layer inside the mold. The samples were removed from the mold and placed on the base of the cell. A plastic membrane was wrapped around the sample using a suction mold. Two rings were placed on the upper and lower caps to seal the sample. Then, the cell chamber was closed and the water taps were opened. Three confining stresses of 100, 200 and 300 kPa were applied to the stabilization samples with 3, 5 and 7% Nano-Typha in three curing times of 1, 7 and 28 days. Three tests were also performed on the base clay. Figure 4 shows an example of the device used in the research. The loading rate for the triaxial tests was conducted at a rate of 0.228 mm/min.

Results

Figure 5 shows the stress-strain diagram obtained from the unconfined compressive strength test for Khavaran clay. In this diagram, the maximum stress value represents the unconfined compressive strength at optimum moisture content. For clay, this value is 50 kPa. In addition, the failure strain is 1.2% and the elasticity modulus (E_{50}) is 10 MPa.

The stress-strain diagram for the stabilized soil with 3% Typha at the curing time of 28 days is shown in Fig. 6. For soil stabilized with Typha, the maximum stress value (as shown in in this diagram) is 75 kPa, the failure strain is 1.2% and the elasticity modulus (E_{50}) is 11 MPa.

Figure 7 shows the stress-strain curve for the samples stabilized with 3% Nano-Typha particles after 1, 7 and 28 days of curing. The unconfined compressive strength of the base clay increased by 12 times after 28 days of curing by adding 3% of nano-particles. The strength increase and the failure strain decrease made the slope of the curve increase and the elasticity modulus increase as well. Moreover, the soil behavior became more

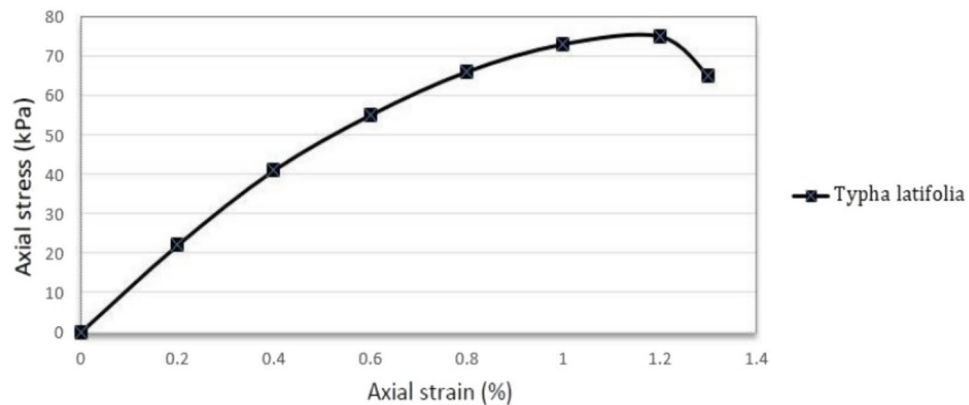


Fig. 6. The stress–strain curve of stabilized clay with 3% Typha in the curing time of 28 days.

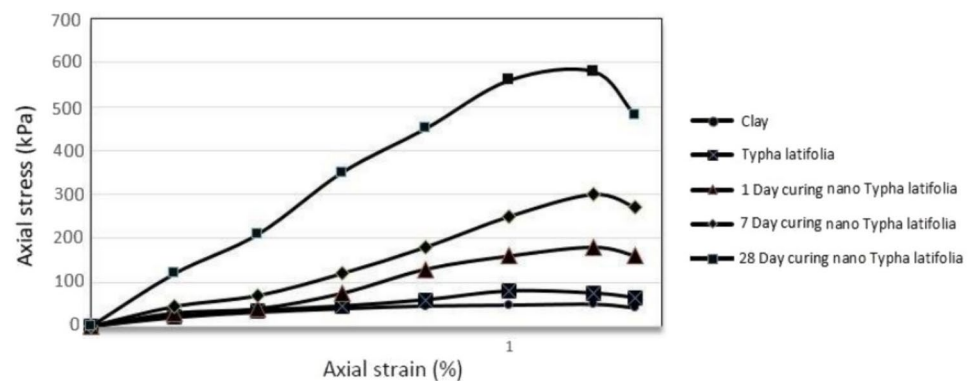


Fig. 7. The stress–strain curve of samples stabilized with a 3% solution of Typha Nanoparticles at different times of curing.

brittle and the strain at failure decreased with the increase of curing time. This was due to the completion of the reaction between nano-particles and soil and the hardening behavior of the soil. The results show the great effect of Nano-Typha solution on the soil compressive strength.

Figure 8 illustrates the effect of Nano-Typha solution on the compressive strength of soil samples with different percentages (3%, 5%, and 7%) and curing times (1, 7, and 28 days). The results show that increasing the Nano-Typha percentage from 3 to 5% enhances the soil's uniaxial resistance by 3.6 to 4.6 times for all curing times. The highest compressive stress (600 kPa) is achieved by the sample with 7% Nano-Typha after 28 days of curing. The increased reactivity and surface area of Nano-Typha particles, as well as their ability to fill the soil pores, contribute to the strength enhancement. The reduction in failure strain and the increase in elastic modulus indicate that the percentage of Nanoparticles affects the deformation behavior of the soil.

Figures 9 and 10 compare the compressive strength ratio of soil samples stabilized with Nano-Typha solution to those without stabilization and with conventional stabilization after 28 days of curing. The results reveal that Nano-Typha solution enhances the uniaxial compressive strength of the soil in the early stages of curing, which is a crucial factor for the soil performance. This is due to the chemical reactions between Nano-Typha particles and soil that occur over time. The presence of Nano-Typha particles in the clay pores and voids reduces the permeability, increases the interlocking between grains and alters the soil texture. Nano-Typha particles are extremely small and can fill the tiny spaces (pores and voids) within the clay matrix. When these nanoparticles are introduced into the soil, they occupy the spaces between the soil particles, effectively blocking the pathways through which water would normally flow. This process is known as pore filling.

Figure 11 shows the failure strain of soil samples stabilized with different percentages of Nano-Typha solution (3, 5, and 7%) at different curing times (1, 7, and 28 days). The results indicate that the failure strain decreases with increasing Nano-Typha percentage and curing time. For example, at 7 days of curing, the failure strain for the samples with 3, 5, and 7% Nano-Typha is 1.25, 1.2, and 1.1, respectively. At 28 days of curing, these values drop to 1.2, 1, and 0.9, which is a 0.75-fold reduction compared to the unstabilized soil. This reduction also implies a transition from a ductile to a brittle behavior of the soil in the stress–strain curve. The higher percentage of Nanoparticles increases the stiffness and brittleness of the stabilized soil and reduces the strain at peak strength for all curing times. Nanoparticles can increase both the stiffness and brittleness of stabilized soil.

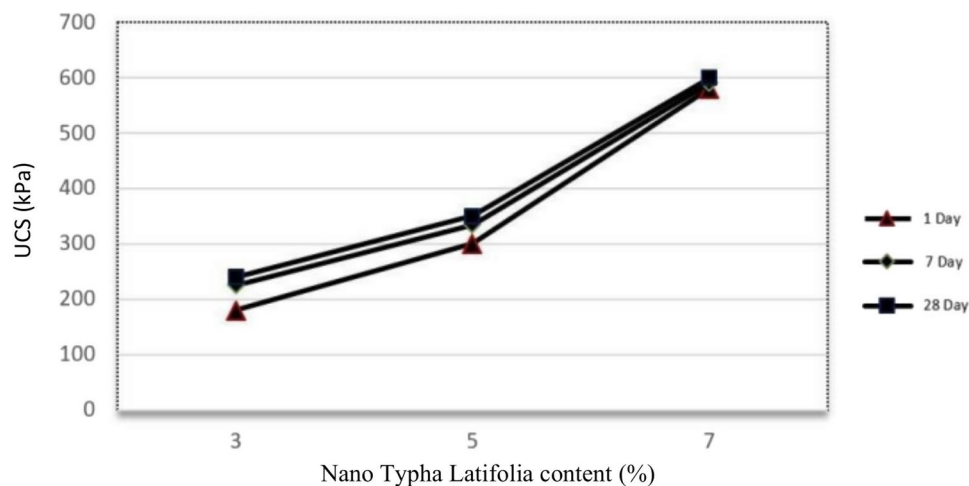


Fig. 8. Compressive strength of the samples stabilized with 3, 5 and 7% of Nano Typha solution in curing times of 1, 7 and 28 days.

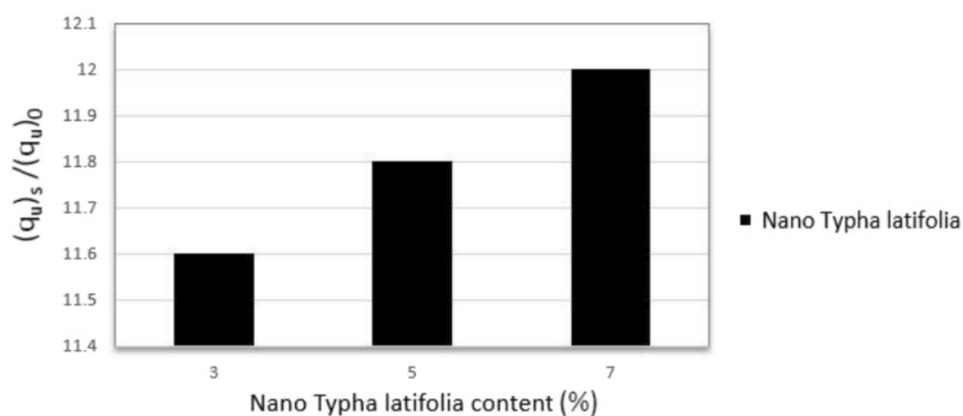


Fig. 9. The ratio of the compressive strength of samples stabilized with Nano-Typha solution in 28-day curing time to unstabilized soil.

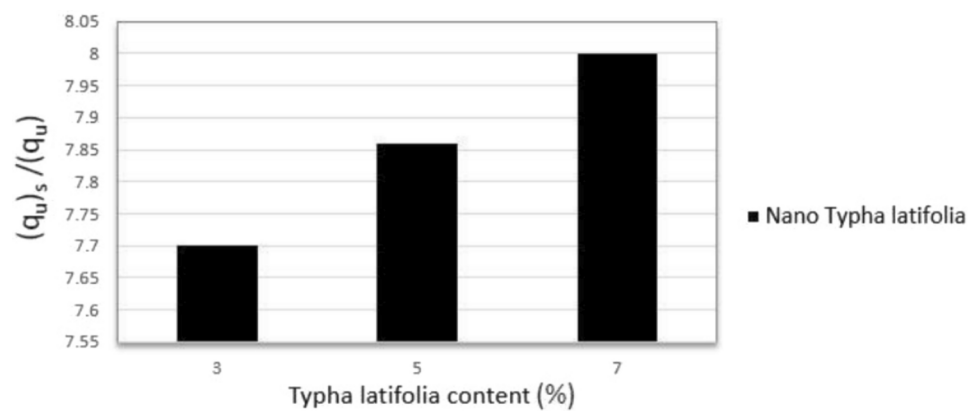


Fig. 10. The ratio of the compressive strength of samples stabilized with Nano-Typha solution in 28-day curing time to the stabilized sample with Typha.

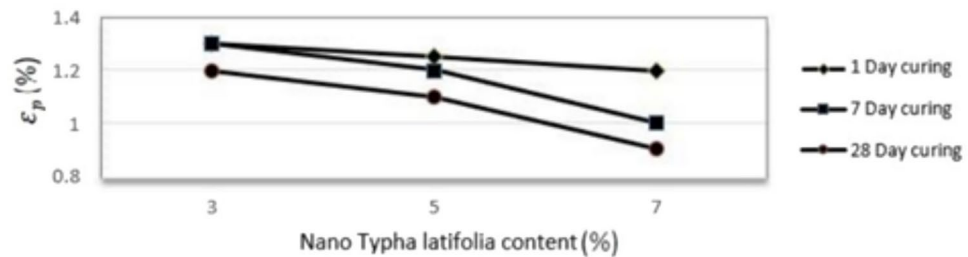


Fig. 11. Values of failure strain in samples stabilized with 3, 5 and 7% of Nano-Typha solution in curing times of 1, 7 and 28 days.

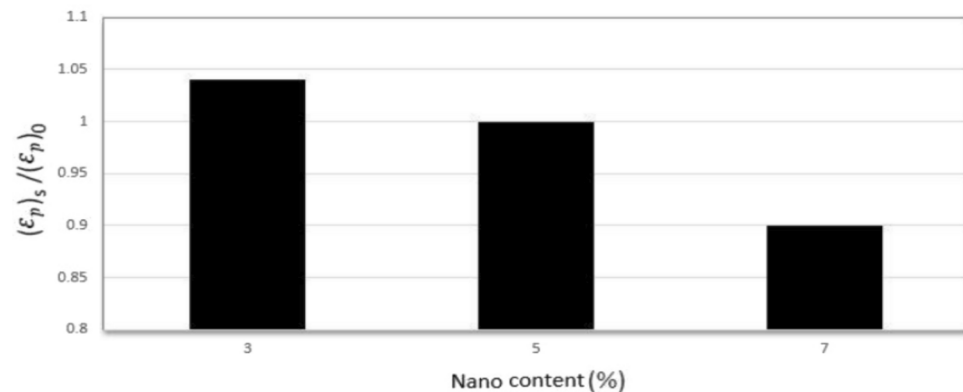


Fig. 12. Failure strain ratio in 7 days samples, stabilized with different percentages of Nano-Typha solution to unstabilized soil.

due to their unique properties and interactions with the soil matrix. When nanoparticles are added to soil, they fill the voids between soil particles, leading to a denser and more compact structure.

Figure 12 depicts the failure strain ratio of soil samples stabilized with Nano-Typha solution to those without stabilization. The figure demonstrates that the failure strain of the stabilized samples decreases relative to the unstabilized samples. For instance, at 7 days of curing, the failure strain ratio for the samples with 3, 5, and 7% Nano-Typha is 1.25, 1.2, and 1.1, respectively. At 28 days of curing, these ratios reduce to 1.2, 1, and 0.9, which is a 0.75-fold decrease compared to the unstabilized soil. This decrease also implies a transition from a ductile to a brittle behavior of the soil in the stress–strain curve. The higher percentage of Nanoparticles increases the stiffness and brittleness of the stabilized soil and reduces the strain at peak strength for all curing times.

Figure 13 presents the elastic modulus of soil samples stabilized with different percentages of Nano-Typha solution (3, 5, and 7%) after 28 days of curing. The elastic modulus is calculated from the ratio of stress change to strain change in the elastic region. The results show that the elastic modulus increases with increasing Nano-Typha percentage. The values are 30, 30.5, and 30.7 MPa for the samples with 3, 5, and 7% Nano-Typha solution, respectively. These values are higher than those of the unstabilized soil. Figure 14 compares the elastic modulus ratio of the stabilized samples to the unstabilized soil. The figure indicates that the elastic modulus ratio increases with increasing Nano-Typha percentage. After 28 days of curing, the elastic modulus ratio for the samples with 3, 5, and 7% Nano-Typha is 3, 3.05, and 3.07, respectively. Figure 15 illustrates the effect of curing time on the elastic modulus ratio. The figure reveals that the elastic modulus ratio slightly increases with increasing curing time for all Nano-Typha percentages. After 28 days of curing, the elastic modulus ratio for the samples with 3, 5, and 7% Nano-Typha is 2.7, 2.77, and 2.8, respectively. This increase reflects a higher elastic modulus and a more brittle behavior at longer curing times. The addition of Nano-Typha solution reduces the porosity and improves the bonding between the soil particles due to the increased specific surface area. This results in a denser and more brittle soil behavior.

Figure 16 displays the stress–strain behavior of the on-site soil under three different confining stresses (100, 200, and 300 kPa). The figure shows that the deviator stress increases with increasing axial strain until it reaches its peak value. The peak deviator stress values for the on-site soil at confining stresses of 100, 200, and 300 kPa are 65, 80, and 90 kPa, respectively.

Figure 17 depicts the stress–strain behavior of soil stabilized with 3% Micro-Typha solution under three different confining stresses (100, 200, and 300 kPa). The peak deviator stress values for the soil stabilized with 3% Micro-Typha solution at confining stresses of 100, 200, and 300 kPa are 140, 180, and 210 kPa, respectively.

Figure 18 illustrates the stress–strain behavior of soil stabilized with 3% Nano-Typha solution under three different confining stresses (100, 200, and 300 kPa). The figure shows that the deviator stress difference between

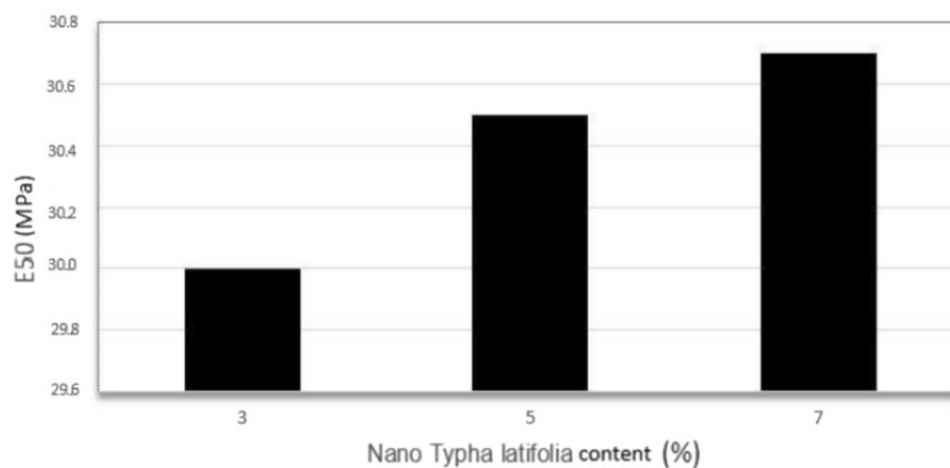


Fig. 13. The effect of different percentages of Nano Typha solution during 28-day curing time on elastic modulus.

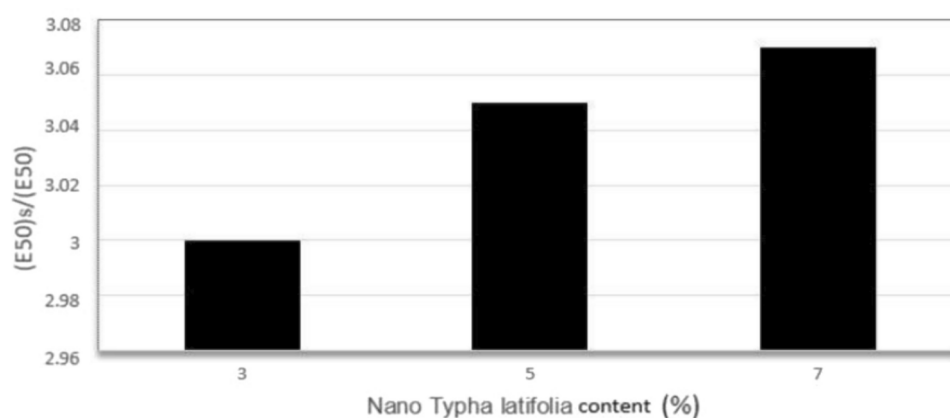


Fig. 14. The ratio of the elastic modulus of the samples stabilized with Nano-Typha solution to the unstabilized soil with different percentages in the curing time of 28 days.

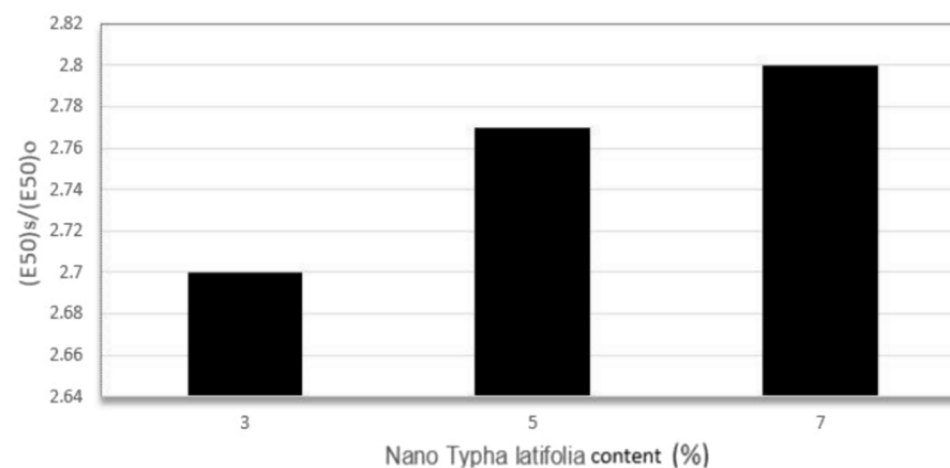


Fig. 15. The ratio of the elastic modulus of the samples stabilized with Nano-Typha solution to the soil stabilized with Typha with different percentages in the curing time of 28 days.

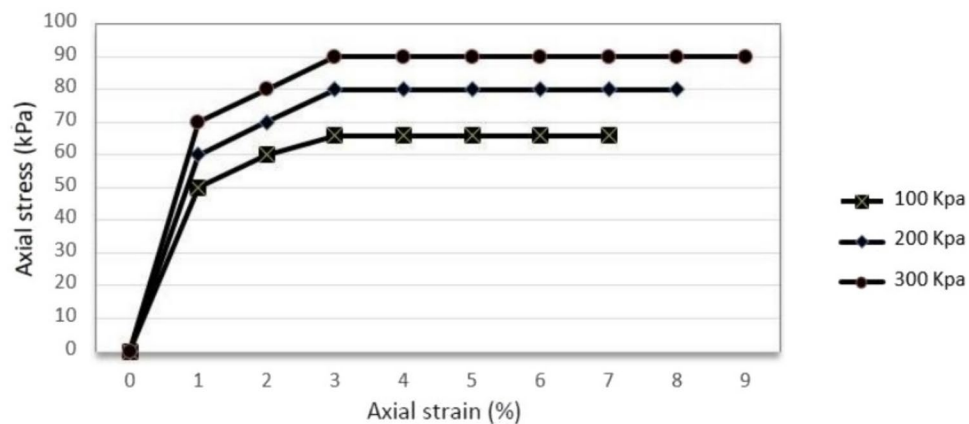


Fig. 16. The stress–strain diagram of the soil on site in unstabilized conditions in 3 confining stresses.

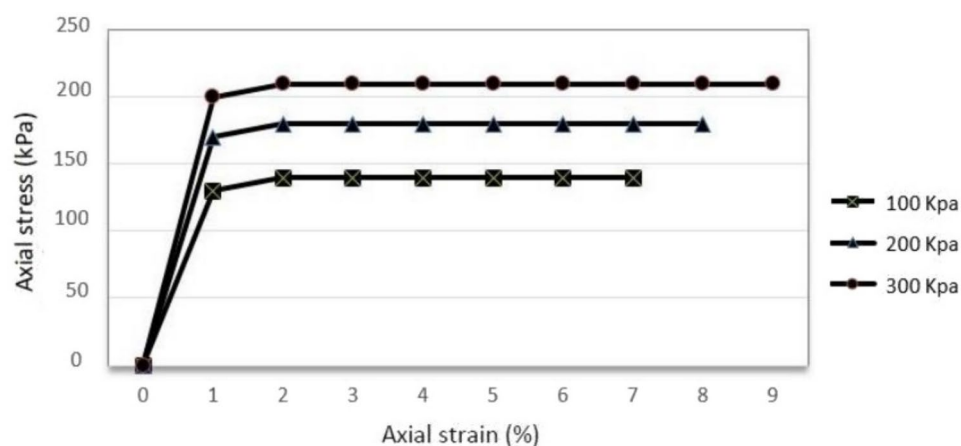


Fig. 17. Stress–strain diagram of soil stabilized with Typha in unstabilized conditions in 3 confining stresses.

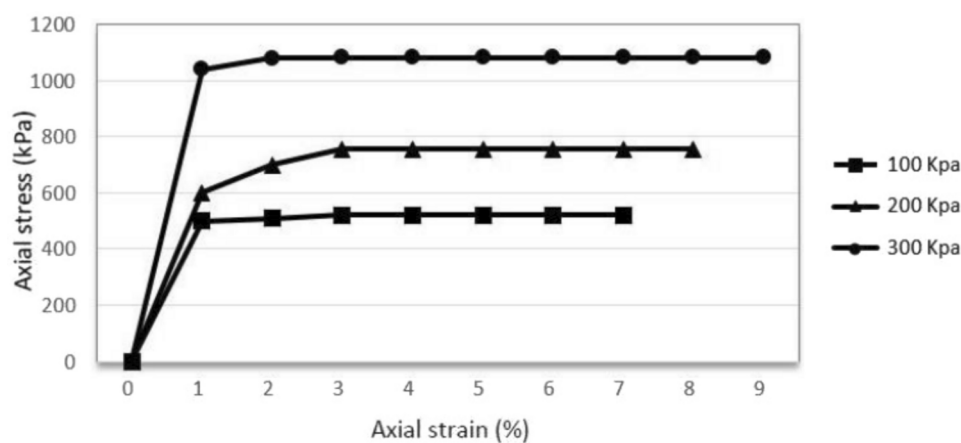


Fig. 18. Stress–strain diagram of the stabilized sample with 3% Nano-Typha.

the unstabilized and stabilized soil increases with increasing confining stress. The deviator stress difference values for the soil stabilized with 3% Nano-Typha solution at confining stresses of 100, 200, and 300 kPa are 520, 755, and 1082 kPa, respectively. The figure also indicates that the failure behavior of the soil changes from softening to hardening as the confining stress increases.

Material	3%	5%	7%
<i>Typha latifolia</i>	11.7	11.8	12
Nano <i>Typha latifolia</i>	31	31	32

Table 2. Internal friction angle values for the soil on site and stabilized samples with different percentages of types of Nanomaterials and non-Nanomaterials.

Material	3% (kPa)	5% (kPa)	7% (kPa)
<i>Typha latifolia</i>	25.6	25.6	26
Nano <i>Typha latifolia</i>	40	40	55

Table 3. Cohesion values for the soil on site and stabilized samples with different percentages of types of Nanomaterials and non-Nanomaterials.

Element	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂
Wt %	2.153	2.309	< <	10.997	5.448	6.810	29.936	19.866	< <
Element	Fe ₂ O ₃	V ₂ O ₅	MnO	Cr ₂ O ₃	Ni	Zn	Sr	Y	Pb
Wt %	11.050	0.917	1.134	< <	> >	–	–	–	–
Element	Ba	Zr	Cl	Co	Ce	Mo	Nb	Cu	Sn
Wt %	–	–	9.379	–	–	–	< <	< <	–

Table 4. XRF test results before the production proces of Nano-Typha.

Element	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂
Wt %	2.153	2.309	< <	10.997	5.448	6.810	29.936	19.866	< <
Element	Fe ₂ O ₃	V ₂ O ₅	MnO	Cr ₂ O ₃	Ni	Zn	Sr	Y	Pb
Wt %	11.050	0.917	1.134	< <	> >	–	–	–	–
Element	Ba	Zr	Cl	Co	Ce	Mo	Nb	Cu	Sn
Wt %	–	–	9.379	–	–	–	< <	< <	–

Table 5. XRF test results after the production proces of Nano-Typha.

The internal friction angle and cohesion parameters of the on-site soil are calculated from the deviator stress values obtained from the stress–strain curves for three confining stresses (100, 200, and 300 kPa). The values are 5° and 24.8 kPa for the on-site soil. Table 2 compares the internal friction angle values for the on-site soil and the soil samples stabilized with Typha and Nano-Typha. The results show that Nano-Typha has a significant effect on increasing the soil friction angle up to 32°. The soil sample stabilized with Nano-Typha has a 2.6-fold higher friction angle than the one stabilized with Typha.

Table 3 shows the cohesion values at the failure point of soil samples stabilized with different percentages of Nano-materials. The on-site soil has a cohesion value of 24.8 kPa. The cohesion values of the stabilized samples increase with increasing Nano percentage and reach 55 kPa for the sample with 7% Nano-Typha. The samples stabilized with Nano-Typha have higher cohesion values than those stabilized with Typha. This is attributed to the chemical reaction between Nanoparticles and clay. The addition of Nano-Typha increases the specific surface area and creates more bonds and a complex structure in the soil, which enhances the soil cohesion. The soil sample stabilized with Nano-Typha has a twofold higher cohesion than the one stabilized with Typha.

XRF analysis

X-ray Fluorescence Spectroscopy (XRF) is a technique for elemental identification that is useful for some industries such as cement and steel because of its high speed. In the XRF laboratory, an unknown sample is irradiated with X-rays and emits secondary X-rays due to atomic excitation. The secondary X-rays can be analyzed by either the Wavelength Dispersive Spectroscopy (WDS) method or the Energy Dispersive Spectroscopy (EDS) method to identify the element or elements in the sample. The XRF tests can report the percentages of elements from sodium to uranium in the range of 0.001–99.99%. To ensure that the soil was not contaminated by the wear of metal and ceramic balls during the Nano production process, an XRF test was performed on 2 g of soil after hours of Nano production. The results of the XRF test are shown in Tables 4 and 5.

The XRF test results of the sample before and after the Nano production process show that no changes have occurred in the constituent elements of the powder.

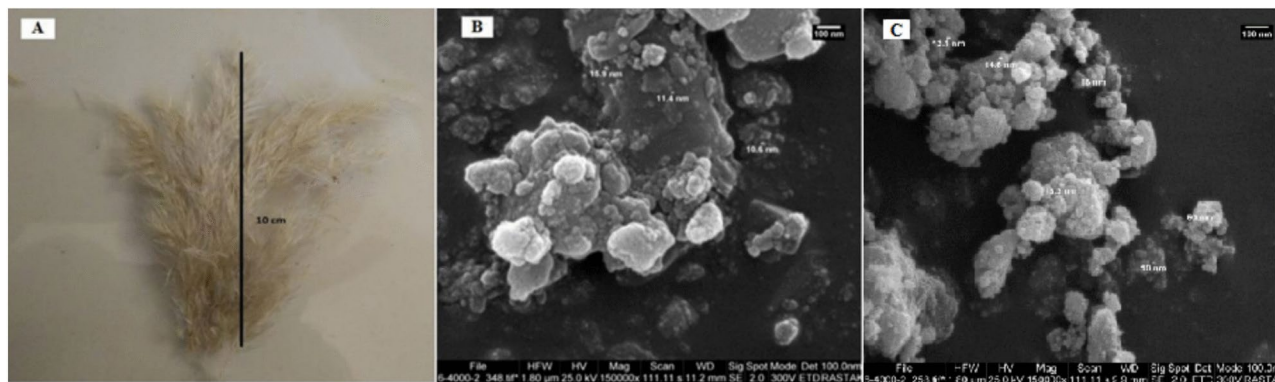


Fig. 19. Typha plant (a) before and (b, c) after conducting the process of Nano production.

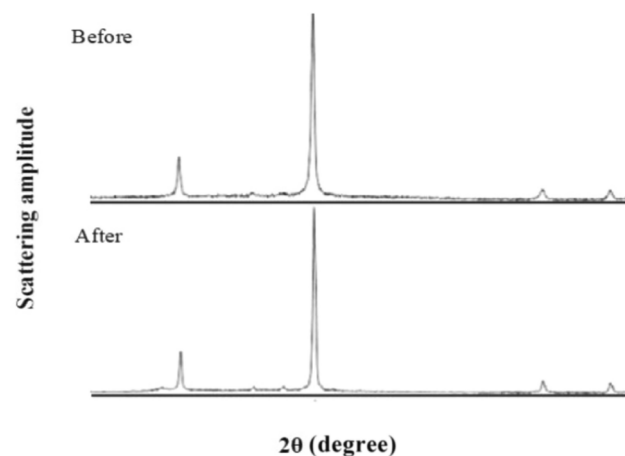


Fig. 20. XRD diagram before and after the process of Nano-Typha production.

SEM analysis

Scanning Electron Microscopy (SEM) is a technique for imaging and characterizing the surface features and morphology of samples. This technique uses electrons to record the image, which have a much higher magnification power than light microscopes. They can produce images with magnifications up to several hundred thousand times. Figure 19 shows the Typha plant before and after the Nano production process. The Typha plant has a fibrous and irregular texture. After the Nano production process, the Typha plant is converted into nanoparticles with sizes ranging from 5 to 130 nm, as shown in Fig. 19. The SEM images in Fig. 19a depict the fibrous and irregular texture of the Typha plant before the process, while Fig. 19b illustrates the formation of nanoparticles after the process. Figure 5 shows the results of the SEM test to verify the particle size distribution.

XRD analysis

X-ray Diffraction (XRD) analysis is a technique for studying the crystalline structures of materials. This technique uses X-ray diffraction and Bragg's law to produce a unique spectrum for each compound. The XRD test can identify the composition and formula of any unknown sample in the XRD laboratory. To ensure that the soil was not contaminated by the wear of metal and ceramic balls during the Nano production process, an XRD test was performed on 2 g of soil after hours of Nano production. Figure 20 shows the XRD diagrams before and after the process of Nano-Typha production. The XRD diagram before the process indicates the initial crystalline structure of the soil, while the diagram after the process shows any changes in the crystalline structure due to the Nano production. This comparison helps verify that the soil's composition remains unchanged and free from contamination.

Conclusion

This research investigated the production of nanoparticles from the Typha plant and their effect on Khavaran clay. Key findings include:

1. Adding 3% Nano-Typha or Typha increases the unconfined compressive strength significantly after 1 day of curing, with Nano-Typha showing a 3.6-fold increase and Typha a 1.5-fold increase.

2. Nano-Typha reduces the strain at failure, with 7% Nano-Typha solution showing a 0.75-fold lower strain after 28 days of curing compared to unstabilized soil.
3. Soil stabilized with 7% Nano-Typha solution exhibits maximum deviator stresses of 606, 985, and 1273 kPa at confining stresses of 100, 200, and 300 kPa, respectively. Cohesion and friction angle parameters are significantly higher, with Nano-Typha showing 2.22 and 6.3 times increases, respectively, and Typha showing 2.3 and 1.01 times increases.
4. Elastic modulus increases with the addition of Nano-Typha or Typha, up to 2.8 times for Nano-Typha and 1.1 times for Typha.

Data availability

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

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Author contributions

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Declarations

Competing interests

The authors declare no competing interests.

Additional information

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